Thermal Decomposition of 3-Nitro-1,2,4-Triazole-5-One (NTO) and Nanosize NTO Catalyzed by NiFe₂O₄

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Thermal decomposition of 3-nitro-1,2,4-triazole-5-one (NTO) and nanosize NTO catalyzed by NiFe₂O₄

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Abstract

Nanosize Nickel ferrite (NiF) was synthesized by the co-precipitation methods and its effect as a 5 % by mass additive was studied on the thermal decomposition of micrometer and nanometer size NTO. In the presence of 5 % NiF additive, the thermal decomposition peak temperature of NTO was decreased from 276.36 to 260.18 °C and that of nano NTO was decreased from 261.38 to 258.89 °C ($\beta=10$ °C min⁻¹). The kinetics parameters confirms the catalytic activity of NiF for the thermal decomposition of NTO, and nNTO as the parameters such as activation energy (NTO= $\sim$ 25.45 % and nNTO= $\sim$ 45.94 % decrement), and pre-exponential factor (NTO= $\sim$ 21.94 % and nNTO= $\sim$ 43.12 % decrement) were decreased when 5 % NiF additive was added to NTO, and nNTO. The rate of the decomposition process was increased in the presence of 5 % NiF catalyst, indicating the faster thermal decomposition of both NTO, and nNTO in the presence of nickel catalyst.

Keywords: 3-nitro-1,2,4-triazol-5-one (NTO), Nickel ferrite, Catalyst, Decomposition

Introduction

High energetic material (HEMs) are special class of chemical components that are used for the applications such as construction, rocket propellants, missiles, gunpowder, mining, etc. as HEMs release immense energy and gases that can be utilized to thrust an object or destroy a target. The examples of such HEMs includes ammonium perchlorate, ammonium nitrate, 1,3,5,7-tetranitro-1,3,5,7-tetraazacyclooctane (HMX), 1,3,5-trinitro-1,3,5-triazinane (RDX), 3-nitro-1,2,4-triazole-5-one (NTO) etc.[1] NTO (Impact sensitivity= $57.5 \text{ H}_{50} \text{ cm}^{-1}$) is one of the most potential candidate for replacing the otherwise sensitive HEMs such as HMX (Impact sensitivity= $22.4 \text{ H}_{50} \text{ cm}^{-1}$), RDX (Impact sensitivity= $25.6 \text{ H}_{50} \text{ cm}^{-1}$) etc. as it is highly insensitive to external stimuli.[2,3] However, research have been carried out to improve the thermal decomposition performance of NTO which can directly influence the burning and combustion performance of explosive compositions. The thermal properties of NTO can be improved by use of (i) additives[4,5], (ii) nanosizing of NTO[6,7], (iii) and/or Forming the co-crystals with other HEMs such as HMX[2]. The various literatures[8–10] reported that metal oxide (MO) based nanomaterials can significantly improve the thermal decomposition of HEMs such as AP, HMX, RDX, etc. due to the smaller size and large surface-to-volume ratio of MOs. i.e., Wei et al.[11] reported that Bi_2WO_4 nanomaterial with particle size of 40 nm show better results on the thermal decomposition of HEMs such as RDX, HMX, ammonium perchlorate and double base propellants as well as burning rate of double base propellant compare to 100 nm size Bi_2WO_4 . MOs can facilitate the electron/proton transfer during the decomposition of HEMs as the metal such as transition metals can exist in variety of oxidation states and absorb the gaseous generated during the decomposition catalyzing the intermediate reactions. However, only few studies have reported the effect of transition metal based additives on the thermal decomposition of NTO. Study of Prabhakaran et al.[5] show effect of various TMOs as a 5 % by mass additive and found that TMOs can be used to improve the exothermic peak temperature of NTO, however, the kinetic parameters were not studied which plays an important role in determining the catalytic activity. Hanafi et al.[4] studied that incorporating the 2D energetic hybrid crystals containing metals such as Zn, and Co not only decreases the peak temperature of an exothermic curve of NTO, but also decreases high the activation energy of NTO with enhanced heat of decomposition. The previous studies have reported that AB_2O_4 type metal oxides such as MFe_2O_4 (where $\text{A}=\text{M}=\text{Co}^{2+}$, Cu^{2+} , Ni^{2+} , etc. $\text{B}=\text{Fe}$, Co etc.) imparts good catalytic activity for decreasing the thermal decomposition temperature and activation energy of the decomposition of HEMs such as ammonium perchlorate, ammonium nitrate etc. along with enhancing the burning rate of the corresponding solid rocket propellants.[12,13] The

ease of synthesis, utilization of less toxic and cost effective chemicals, less energy consumption during the synthesis, and magnetic separation of ferrites gives them advantage over other costly catalysts/additives. Previously an article[14] suggested that Metal salts of NTO such as Fe-NTO, and Ni-NTO can efficiently improve the burning rate of Composite propellants.

In the present study, we have reported the application of 5 % by mass nanosize nickel ferrite (NiFe_2O_4 or NiF) catalyst for improving the thermal decomposition and kinetic parameters such as activation energy, pre-exponential factor of NTO, and nanosize NTO (nNTO). The TG-DSC data was used for the evaluation of the effect of catalyst on the decomposition of NTO, and nNTO. Three isoconversion methods namely Flynn-Wall-Ozawa (FWO), Kissinger-Akahira-Sunose (KAS), and Starink method were used for calculating the kinetic parameters.

Materials

Metal nitrate salts and sodium hydroxide used in the present work were of acquired from local providers and used as such without further purification. NTO was synthesized in lab using previously reported method.[15] Nanosize NTO was prepared using solvent (tetrahydrofuran)-antisolvent (n-hexane) approach.[16]

Synthesis

Wet chemical precipitation method was used to synthesize nanosize NiF. Briefly, nitrate salts of $\text{Ni}^{2+}:\text{Fe}^{3+}$ in 1:2 mole ratio were mixed in 100 mL distilled water. To this mixture 2 M NaOH was added under magnetic stirring to obtain the black precipitates of corresponding hydroxide salts [$\text{Ni}(\text{OH})_2$ and $\text{Fe}(\text{OH})_3$]. After completion of the precipitation (pH~11-12), the precipitation were filtered, washed with hot water to wash away impurities, and dried in an hot air oven at 60 °C. Finally, the calcination of the precipitates at 500 °C for 5 hours results in a muffle furnace yields nanosize NiF.

NiF nanocatalyst was mixed with NTO and nNTO in 5:95 mass ratio to produce mixture of NTO+NiF, and nNTO+NiF, respectively. The four compositions (NTO, nNTO, NTO+NiF, and nNTO+NiF) were used for the decomposition study using TG-DSC data.

Characterization

Structure of NiF was confirmed using powder X-ray diffraction (XRD, Ultima IV Powder X-Ray Diffractometer instrument equipped with a Cu K α radiation source; $\lambda=1.5406$ Å) and Raman analysis (JobinYvon Horiba LabRam, HR800 laser source; $\lambda= 532$ nm).

Catalytic activity

Thermal decomposition of NTO compositions was evaluated using TG-DSC (Perkin Elmer, STA 8000) at three linear heating rates (5, 10, and 15 °C min⁻¹ or K min⁻¹) from room temperature to 400 °C temperature under inert nitrogen atmosphere in a platinum pan. The obtained data was used to calculate the decomposition kinetic parameters of four NTO compositions.

Result and Discussions

The XRD patterns of NiF were compared with COD database file (5910064; Reference code: 96-591-0065) to confirm the formation of ferrite. The XRD spectrum of NiF is provided in Figure 1-a. NiF exist patterns at 2θ values 29.40°, 35.66°, 38.94°, 43.34°, 47.76°, 57.34°, and 63.12° values corresponding to (220), (311), (222), (400), (331), (422), (511), and (440) planes, respectively. NiF has a cubic structure with F d -3 m (227) space group. The XRD data was used to calculate the lattice parameter (a , eq. 1), cell volume (a^3 ; V), and X-ray density ($\rho_{X\text{-ray}}$, eq. 2). William-Hall equation (eq. 3) was used to evaluate the lattice strain and crystalline size of NiF. Interception, and slope of plot of $4\sin\theta$ on x-axis against $\beta\cos\theta$ on y-axis gives the value of crystalline size and lattice strain, respectively (Figure 1-b).

$$a = d\sqrt{h^2 + k^2 + l^2} \quad (1)$$

$$\rho_{x\text{-ray}} = \frac{8M}{N_A a^3} \quad (2)$$

$$\beta\cos\theta = \frac{k\lambda}{D} + 4\epsilon\sin\theta \quad (3)$$

Where, a is the lattice parameter (Å), d is the inter-atomic spacing (Å), and h , k , and l are the miller indices. M represents the molar mass of NiF (234.38 g mol⁻¹), N_A is the Avogadro's number (6.022×10²³ mol⁻¹), k is the constant value of which is usually taken as 0.9, λ is the wavelength of the radiation source (0.15406 nm for Cu K- α), D is the crystalline size (nm), ϵ is the lattice strain, β is the full width at half maxima of the given XRD peak, and θ is the Bragg's angle ($2\theta/2$ in degree). The parameters calculated from the powder XRD data for

NiF are depicted in Table-1. The lattice parameters was close to the previously reported values, and so was the X-ray density. The general trend was found from the comparison that as the X-ray density was increased, the size of NiF was also increased as the larger size particles require less volume for the same mass of the substance. However, not all results were according to this trend.[17]

Figure 1. (a) XRD patterns and (b) William-Hall plot of NiF

Table 1. Calculated parameters from XRD data of NiF

Lattice constant/Å (a=b=c)	Angle ($\alpha=\beta=\gamma$)/degree	Cell Volume/Å ³	X-ray Density/g cm ⁻³	Average crystalline size/nm	Lattice-strain (ϵ)	Ref.
8.32	90°	572.52	5.40	16.90	0.0129	Present work
8.23		557.00	5.59	22.00	-	[18]
8.33		578.63	5.38	15.60	-	[19]
8.34		579.10	5.41	14.82	-	[17]

The Raman spectrum (Figure S 1) of NiF confirms the formation of spinel ferrite. NiF has five Raman active modes. The five Raman active modes for NiF were observed at 206.40, 317.49, 478.21, 555.41, and 690.01 cm^{-1} corresponding to $F_{2g}(1)$, E_g , $F_{2g}(2)$, $F_{2g}(3)$, and A_{1g} active modes, respectively. The Raman peaks of NTO, and nNTO with and without 5 % by mass NiF additive exhibits similar pattern. The broadening of the Raman peaks was observed when NiF was added to NTO, and nNTO. The broadening could be because of the nanosize of NiF as peaks are broadened in nanosize material compare to micron size material.[20]

Kinetics study

Three heating rates (5, 10, and 15 $^{\circ}\text{C min}^{-1}$) were used to evaluate thermo kinetic parameters. DSC peak area was used to evaluate extent of conversion (α) at various heating rates using Eq. 1. Where, dH/dt represents heat flow. Further, three isoconversion integral methods FWO (Eq. 2)[21,22], KAS (Eq. 3)[23,24], and Starink (Eq. 4)[25] were used to evaluate activation energy (E_a), and pre exponential factor (A) at varying extent of conversion.

$$\alpha = \frac{\int_0^t \left(\frac{dH}{dt} \right) dt}{\int_0^{t_a} \left(\frac{dH}{dt} \right) dt} = \frac{\Delta H}{\Delta H_{total}} \quad (1)$$

$$\ln \beta = \ln \left(\frac{0.0048 A E_a}{g(\alpha) R} \right) - 1.0516 \frac{E_a}{RT} \quad (2)$$

$$\ln \left(\frac{\beta}{T^2} \right) = \ln \left(\frac{AR}{g(\alpha) E_a} \right) - \frac{E_a}{RT} \quad (3)$$

$$\ln \left(\frac{\beta}{T^{1.92}} \right) = \ln \left(\frac{AR^{0.92}}{E_a^{0.92}} \right) - 0.312 - 1.0008 \frac{E_a}{RT} \quad (4)$$

Where, β , R, T, and $g(\alpha)$ represents heating rate ($^{\circ}\text{C min}^{-1}$), universal gas constant (8.314 $\text{J K}^{-1} \text{mol}^{-1}$), Temperature (K), and integral reaction model, respectively. E_a is evaluated from the slope and A from the intercept of the Plots of $\ln \beta$, $\ln(\beta/T^2)$, and $\ln(\beta/T^{1.92})$ against $1000/T$ (K^{-1}). dH/dt is the DSC heat flow.

Thermal decomposition

The TG curve of four NTO compositions is given in Figure 2. The decomposition of NTO takes place in single step at $\sim 259^\circ\text{C}$ and that of nNTO takes place at $\sim 256^\circ\text{C}$. When 5 % by mass NiF is mixed, the Decomposition of both NTO, and nNTO occurs in two steps. The first mass loss occurs $\sim 200^\circ\text{C}$ with a mass loss of 5.17 % in case of NTO+NiF and 6.98 % in case of nNTO+NiF. The second mass loss for both NTO+NiF, and nNTO+NiF occurs between ~ 200 - 260°C with mass loss of 63.75 % and 64.08 %, respectively. The thermal stability order was: $\text{NTO} > \text{nNTO} > \text{NTO} + \text{NiF} > \text{nNTO} + \text{NiF}$. The stability of both NTO, and nNTO was decreased in the presence of NiF catalyst. Although the initial decomposition step of NTO is not clear, it is believed to proceed through H-abstraction (from N-H) and cleavage of C-NO₂ bond.[26] Hence, it is proposed that NiF can abstract H from N-H bond and facilitate the cleavage of C-NO₂ bond. NiF can adsorb the gases released during the decomposition of NTO catalyzing the decomposition reaction.

Figure 2. TG curve of NTO, and nNTO with and without 5% by mass NiF catalyst ($\beta=10^\circ\text{C min}^{-1}$)

The DSC curve (Figure 3) of NTO show single exothermic peak corresponding to decomposition of NTO at ~ 250 - 280°C with 276.36°C peak temperature. In the presence of NiF catalyst, the DSC curve of NTO shifts to lower temperature (i.e., ~ 240 - 270°C) with a

peak temperature of 260.18 °C. The decrement in the peak temperature of the high temperature exothermic curve was 16.18 °C, the decrement is good as unlike ammonium perchlorate, NTO is not highly affected by the presence of additives.[5] Similarly, The exothermic DSC curve of nNTO was decreased by 2.49 °C. The effect of NiF on the DSC curve of nNTO was not as great as NTO, this could be because nNTO's decomposition takes place at lower temperature compare to micron size NTO because of the reduced size. Both NTO+NiF, and nNTO+NiF exhibit similar DSC curves at with slight difference in the decomposition temperature. (i.e., decomposition of nNTO+NiF is ~2 ° lower than NTO+NiF for both exothermic curves). The additional DSC exothermic curve at ~190-210 °C was observed in the presence of NiF additive.

Figure 3. DSC curve of NTO, and nNTO with and without 5% by mass NiF catalyst ($\beta=10\text{ }^{\circ}\text{C min}^{-1}$)

Kinetics parameters

The kinetic parameters of were investigated using change the DSC curve data at 5, 10, and 15 $^{\circ}\text{C min}^{-1}$. The effect of heating rates on the thermal decomposition curve all four samples are depicted in Figure 4. The DSC curves of all compositions were shifted to higher temperature

values with increasing heating rate. i.e., the decomposition of NTO was completed at higher temperature value at higher heating rate. The DSC curves for all samples corresponding to exothermic curve (~ 240 - 280 °C) was observed at all heating rates but the low temperature exothermic curve (NTO+NiF, and NTO+NiF) was found to be almost diminish at higher heating rate (15 °C min^{-1}).

The activation energy of NTO, and nNTO with and without NiF nanocatalyst is given in Table 2 and the variation in the log A and E from conversion value 0.1 to 0.9 at an interval of 0.025 are depicted in Figure 5. The activation energy and pre-exponential factor calculated using FWO method were higher compare to KAS, and Starink method. The activation energy of NTO was high for initial decomposition, however, the activation energy was decreased drastically with progress of the decomposition. In case of nNTO, the activation energy do decrease as the decomposition proceed, but the change was not as drastic as NTO. When NiF catalyst was present, the energy for the initial decomposition was lower, but as the decomposition proceed, the activation energy was increased. i.e., the activation energy of NTO+NiF, and nNTO+NiF after $\alpha=0.5$ was higher than NTO. The average activation energy of NTO was decreased by ~ 99 kJ mol^{-1} , and that of nNTO was decreased by 224.75 kJ mol^{-1} when NiF catalyst was present. In the presence of NiF, the pre-exponential factor of NTO, and nNTO was decreased largely indicating faster decomposition. The rate constant (k) calculated using Arrhenius equation ($k=Ae^{-E/RT}$) also confirms the faster decomposition of NTO, and nNTO in the presence of NiF catalyst. Hanafi et al.[4] applied four energetic e highly energetic coordination polymers of triaminoguanidine-transition metal (Co or Zn) (T-Co, or T-Zn) complexes, with or without graphene oxide (GO)-T for improving the thermal decomposition performance of NTO. 20 % by mass energetic catalysts were able to reduce the DSC peak temperature of NTO up to 13.3 °C, similarly, in another study[5], the lead monoxide was able to decrease the peak temperature of NTO by 8 °C. In the present work NiF was able to reduce the decomposition of NTO by 16.18 °C.

Figure 4. Effect of heating rates on the DSC curve of NTO, nNTO, NTO+NiF, and nNTO+NiF ($\beta=5, 10, 15\text{ }^{\circ}\text{C min}^{-1}$)

Figure 5. Variation in activation energy and pre-exponential factor at different extent of conversion ($\alpha=0.1$ to 0.9 ; interval= 0.025) using FWO, KAS, and Starink method ($\beta=5, 10, 15\text{ }^{\circ}\text{C min}^{-1}$)

Table 2. Kinetics parameters obtained using FWO, KAS, and Starink method using three heating rates ($5, 10$, and $15\text{ }^{\circ}\text{C min}^{-1}$)

Sample	FWO		KAS		Starink		Rate constant (k)/s ⁻¹
	$E_a/\text{kJ mol}^{-1}$	$\log A$	$E_a/\text{kJ mol}^{-1}$	$\log A$	$E_a/\text{kJ mol}^{-1}$	$\log A$	
1			1		1		

NTO	382.37	40.38	393.26	31.85	393.30	32.10	1.83×10^{-8}
NTO+NiF	286.98	31.52	293.17	25.90	293.28	23.26	1.74×10^{-5}
nNTO	465.79	49.35	481.13	44.21	481.10	41.08	2.52×10^{-5}
nNTO+NiF	251.80	28.07	256.21	22.39	256.35	19.81	1.53×10^{-5}

Table 3. Comparison of effect of various additives on DSC peak temperature and activation energy of NTO.

Additive	Additive content/%	$\Delta T/^{\circ}\text{C}^{\dagger}$	Catalytic activity ($E/E^{*,\dagger\dagger}$)	Ref.
NiF	5	16.18	1.341	Present work
T-Co	20	5.7	1.857	[4]
T-Zn	20	13.3	3.000	
GO-T-Co-T	20	7.2	2.053	
GO-T-Zn-T	20	9.5	2.053	

[†]Difference in the DSC peak temperature of NTO with and without catalyst; ^{††} E and E^* is the activation energy of NTO and NTO with catalyst

Conclusions

The synthesized NiF nanoparticles imparted great catalytic activity for the decreasing the thermal decomposition temperature of both nanometer as well as micrometer NTO. The addition of only 5 % by mass NiF nanocatalyst was able to decrease the DSC peak temperature of nNTO by 2.49 °C, and that of NTO by 16.18 °C. The decreased decomposition temperature can make the decomposition NTO faster, and therefore can improve its decomposition performance. The DSC curves of NTO, and nNTO show only one exothermic peak in corresponding to the thermal decomposition (>260°C), however, in the presence of the nanocatalyst, two peaks were observed one peak ~198 °C, and second peak at >250 °C. The decreased temperature of TG curves in the presence of nanocatlayst indicates that the stability of the compositions of NTO, and nNTO containing 5% catalyst is also decreased.

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Competing Interests

The authors have no relevant financial or non-financial interests to disclose.

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